# PATENT ABSTRACTS OF JAPAN

RE REJECTION

(11)Publication number:

10-218849

(43)Date of publication of application: 18.08.1998

(51)Int.CI.

A61K 7/00 A61K 7/48 C07C233/56

(21)Application number: 09-026530

(71)Applicant:

KAO CORP

(22)Date of filing:

10.02.1997

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# (54) GLYCERYL-ETHERIFIED AMIDE COMPOUND AND COMPOSITION OF AGENT FOR EXTERNAL USE CONTAINING THE SAME

(57) Abstract:

PROBLEM TO BE SOLVED: To obtain the subject new compound excellent 1 in skin improving effect, esp. skin moisturizing effect and rough skin remedying effect or the like, also excellent in formulation stability. SOLUTION: This new amide compound is expressed by formula I [R1 and R5 are each a 2-3C alkylene or group of the formula R'-O-R" (R' and R" are each a 2-3C alkylene); R2 and R4 are each a (hydroxy-substituted) hydrocarbon; R3 is a single bond or (1-24C alkyl-substituted) methylenel, e.g. N,N'-di[2,3- dihydroxypropyloxy)ethyl]-N,N'-dihexadecylmalonamide. The compound of formula I is obtained, for example, by the following process: an amino compound of formula II is dissolved in an alcohol-based solvent, the resultant solution is refluxed under heating and agitation in a dry nitrogen gas stream in the presence of an alkali, a halogen compound of the formula R2aX (R2a is a 8-24C hydrocarbon; X is a halogen) is then dripped into the system which is refluxed under heating to carry out a reaction until the generation of carbon dioxide is ceased, the resulting product is reacted with a dialkyldicarboxylic acid of formula III (R6 is a 1-4C alkyl) under heating in a dry nitrogen gas stream, and the reaction product is purified.

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## **CLAIMS**

[Claim(s)]

[Claim 1] The following general formula (1)

[Formula 1]

 $0 \longrightarrow \begin{pmatrix} 0 & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & 0 \end{pmatrix}$ 

(The inside R1 of a formula, and that R5 is the same or the ether linkage expressed with carbon-number 2, alkylene machine [ of 3 ], or general formula R'-O-R" (however, R' and R " are the same or differing a carbon number 2 or 3 alkylene machines) which may differ and may have branching is shown.) R2 And R4 the hydrocarbon group of the saturation of the same or the carbon numbers 8-24 which it differed and the hydroxyl may replace, or an unsaturation -- being shown -- R3 The methylene group which the alkyl group of single bond or carbon numbers 1-24 may replace is shown. The glyceryl ether-ized amide compound expressed.

[Claim 2] The medicine-for-external-application constituent containing a glyceryl ether-ized amide compound according to claim 1.

glyceryl-ether-ized predetermined amide compound this invention persons By finding out excelling in the skin improvement effects, such as the skin moisture maintenance effect of having excelled extremely, and the rough skin improvement effect, and combination stability, and containing this compound It had the above-mentioned skin improvement effect, even if it reduced the kind and amount of a surfactant or an oily medicine further, the stable emulsion was formed, and it found out that the medicine-for-external-application constituent which has the feel which felt refreshed by hypoviscosity more could be obtained, and this invention was completed.

[0010] That is, this invention is the following general formula (1).

[0011]

[0012] (The inside R1 of a formula, and that R5 is the same or the ether linkage expressed with carbon-number 2, alkylene machine [ of 3 ], or general formula R'-O-R" (however, R' and that R " is the same or the carbon number 2 which may differ, or 3 alkylene machines) which may differ and may have branching is shown.) R2 And R4 the hydrocarbon group of the same, the saturation which it differed and the hydroxyl may replace, or an unsaturation -- being shown -- R3 The methylene group which the alkyl group of single bond or carbon numbers 1-24 may replace is shown. The glyceryl ether-ized amide compound expressed and the medicine-for-external-application constituent containing this compound are offered.

[Embodiments of the Invention] Two or more glyceryl machines are introduced and the glyceryl ether-ized amide compound (1) of this invention has the low melting point as compared with the amide alcoholic compound which does not have a glyceryl machine. Moreover, it excels also in self-organization and the combination nature to a medicine-for-external-application constituent improves.

[0014] It sets to the glyceryl ether-ized amide compound (1) of this invention, and is R1. And R5 It is an ethylene, trimethylene machine, propylene machine or 2, and 2'-oxy-diethylene machine, and is R1. R5 You may differ, even if the same. When a glyceryl machine exists in R1 and R5, the melting point falls further and the combination nature to a medicine-for-external-application constituent improves further.

[0015] R2 And R4 It is the hydrocarbon group of the saturation of the carbon numbers 8-24 which the hydroxyl may replace, or an unsaturation, and is R2. R4 You may differ, even if the same. As a hydrocarbon group of the saturation of these carbon numbers 8-24, or an unsaturation, the alkyl or ARUKENIRU machine of the straight chain of carbon numbers 8-24 or branching is desirable, for example, an octyl, a desyl, a dodecyl, tetradecyl, hexadecyl one, octadecyl, DOKOSHIRU, 2-methyl pentadecyl, isostearyl, 2-ethylhexyl, 2-ethyl hexadecyl, hexa decenyl, 2-heptyl undecyl, 9-octadecenyl machine, etc. are mentioned. R2 And R4 \*\*\*\*\* -- especially a desirable hydrocarbon group is an alkyl group of the straight chain of carbon numbers 12-22, or branched chain, for example, a dodecyl, tetradecyl, hexadecyl one, octadecyl, DOKOSHIRU, a methyl branching isostearyl machine, etc. are mentioned Moreover, as a hydrocarbon group of the saturation of the carbon numbers 8-24 which the hydroxyl replaced, or an unsaturation, the alkyl or ARUKENIRU machine of the straight chain which one hydroxyl replaced, or branching is desirable, for example, a 2-hydroxy octadecyl machine, a 2-hydroxy octadecenyl machine, etc. are mentioned. [0016] R3 It is the methylene group which the alkyl group of single bond or carbon numbers 1-24 may replace. As an alkyl group of the carbon numbers 1-24 which can be replaced by the methylene group concerned A methyl, ethyl, a propyl, butyl, a pentyl, a hexyl, a heptyl, An octyl, a nonyl, a desyl, a undecyl, a dodecyl, tridecyl, tetradecyl, Pentadecyl, hexadecyl, neptadecyl, octadecyl, a nona desyl, Heneicosyl, DOKOSHIRU, nona KOSHIRU, a thoria contest chill, isostearyl, Iso heptadecyl, 2-ethylhexyl, 1-ethyl heptyl, 8-heptadecyl, 8-heptadecenyl, 8, a 11-heptadeca diethyl, 2-heptyl undecyl, 9-octadecenyl, etc. are mentioned.

[0017] The following compounds can be illustrated as a glyceryl ether-ized amide compound of this invention. [0018]

[Formula 4]

[0021] [Formula 7]

$$\begin{array}{c} R^{2a} \\ R^{2a$$

[0026] (-- R2in formula a -- the hydrocarbon group of the saturation of carbon numbers 8-24, or an unsaturation -- being shown -- X -- a halogen -- being shown -- R6 -- the alkyl group of carbon numbers 1-4 -- a methyl group is shown preferably and R1 and R3 are the same as the above It is the method expressed with).

[0027] That is, a glyceryl ether-ized amide compound (7-1) is obtained by using as a start raw material the compound expressed with a formula (3-1), making R2aX (4-1) react to this under existence of alkali, and making a dialkyl dicarboxylic acid (6-1) react to the compound (5-1) subsequently obtained.

[0028] A sodium hydrogencarbonate, a potassium hydrogencarbonate, etc. are mentioned as alkali used for the reaction of a compound (3-1) and a compound (4-1). The condensation reaction of these compounds is performed by making it react under heating reflux until it carries out heating reflux, a compound (4-1) is subsequently dropped and generating of a carbon dioxide stops, dissolving a compound (3-1) in alcoholic system solvents, such as ethanol and propanol, and agitating under a dryness nitrogen air current under solid alkali coexistence of a sodium hydrogencarbonate, a potassium hydrogencarbonate, a sodium carbonate, etc.

[0029] Subsequently, the glyceryl ether-ized amide compound (7-1) of this invention is obtained by heating under a dryness nitrogen air current, agitating the obtained compound (5-1), adding a dialkyl dicarboxylic acid (6-1) to this, carrying out a predetermined-time reaction, and refining by the still better known method.

[0030] in addition -- for example [0031]

[Formula 10]

existence as a catalyst, or those solvents for a compound (3-1).

[0037] It can also be made to react without protic solvents, such as alcohols, such as ethanol and propanol, or water, being the optimal, and using these solvents as a catalyst used for the reaction of a compound (3-1) and an epoxy compound (4-2). The condensation reaction of these compounds adds the water or ethanol as a catalyst one to 100% of the weight to a superfluous compound (3-1) first, and it heats it, stirring under a dryness nitrogen air current. As for the heating temperature in this case, it is desirable that it is below the boiling point of the solvent added as a catalyst. Subsequently, heating stirring is carried out until an epoxy compound (4-2) is dropped and a compound (4-2) is consumed.

[0038] Subsequently, the glyceryl ether-ized amide compound (7-2) of this invention is obtained by heating under a dryness nitrogen air current, agitating the obtained compound (5-2), adding the dialkyl dicarboxylic acid (6-1) which may have branching in this, carrying out a predetermined-time reaction, and refining by the still better known method.

[0039] In addition, when a glyceryl ether-ized amide compound (7-2) is unsymmetrical, it is desirable to manufacture a corresponding compound (5-2) separately, and to manufacture a glyceryl ether-ized amide compound (7-2) using these. [0040] When it has the skin improvement effect and blends with a medicine-for-external-application constituent, by the low melting point, the glyceryl ether-ized amide compound of this invention tends to form liquid crystal, and its solubility and stability over a basis are good, and can blend it with a basis stably and easily. Therefore, the medicine-for-external-application constituent containing the glyceryl ether-ized amide compound of this invention can demonstrate improvement in moisture maintenance capacity, the surface deterioration improvement effect, etc. by applying to the skin.

[0041] The medicine-for-external-application constituent of this invention makes the usual basis come to contain the glyceryl ether-ized amide compound of this invention. Therefore, it can blend with the medicine-for-external-application constituent of this invention besides a glyceryl ether-ized amide compound (1), combining arbitrarily the oil content and surfactant which are generally used as a medicine-for-external-application component, a moisturizer, an ultraviolet ray absorbent, a whitening agent, alcohols, a chelating agent, pH regulator, antiseptics, a thickener, coloring matter, perfume, etc.

[0042] The medicine-for-external-application constituent of this invention can be used as the charge of emulsification makeup of various forms, for example, the water type in an oil, or an oil water type, a cream, a makeup milky lotion, face toilet, the charge of oily makeup, a lip stick, foundation, a skin cleaning agent, a tonic, a charge for a haircut, hair rinse, hair treatment, a hair tonic, a hair-fostering agent, etc.

[0043] although especially a limit does not have the loadings of the glyceryl ether-ized amide compound in the medicine-for-external-application constituent of this invention -- 0.001- of total composition -- it is 0.01 - 20 % of the weight preferably 50% of the weight

[0044]

[Example] Although an example is given to below and this invention is further explained to a detail, this invention is not limited to the following examples.

[0045] The synthetic Dimroth condenser of an example 112-(2, 3-dihydroxy propyloxy)-1-hexadecyl aminoethane (5-a), 10mL isobaric dropping funnel, and a mechanical stirrer. 1-amino-2-(2, 3-dihydroxy propyloxy) ethane 3.9g (28.9mmol), ethanol 30mL, and 0.67g (7.9mmol) of sodium hydrogenearbonates were taught to the 5 mouth 50mL round bottom flask which it had, under the dryness nitrogen air current, 80 degrees C was made to carry out heating reflux in an oil bath, stirring, and 1-BUROMO hexadecane 2.2g (7.2mmol) was dropped over 1 hour using the dropping funnel. After making it react under heating reflux over 6 - 8 hours until generating of a carbon dioxide stopped, precipitation of the produced sodium bromide was filtered using the glassfiber filter paper. Reduced pressure distilling off of the ethanol used as a reaction solvent was carried out, the residue was dissolved in toluene 30mL and liquid separation washing (3x50mL) was carried out by ion exchange water. After adding anhydrous sodium sulfate to the organic layer and drying it, it filtered, reduced pressure distilling off of the solvent was carried out, and 7.2g of rough products was obtained. The silica gel column chromatography refined the rough product (expansion solvent chloroform: methanol = 10:1-3:1), and 2-(2, 3-dihydroxy propyloxy)-1-hexadecyl aminoethane (5-a) 2.5g was obtained as a translucent wax-like solid-state. The physical properties of the obtained compound (5-a) are as follows. [0046] 1 H-NMR delta(CDCl3) = 0.88 (3H, t, J= 7Hz), 1.3 (26H, broad s) 1.55 (2H, broad m), 2.7 (2H, t, J= 7Hz) 2.9 (2H, t, J= 5Hz), 3.2 (3H, broad s), 3.55-3.75 (6H, m, including d (delta= 3.65)), 3.8(1H, m) IR(cm-1):3388, 2924, 2856, 1474, 1466, 1330, 1128, 1082, 1050, 728. [0047] 2) It heated to 120 degrees C under the dryness nitrogen air current, teaching and stirring 2-(2, 3-dihydroxy propyloxy)-1-hexadecyl aminoethane (5-a) 1g (2.8mmol) to synthetic 20mL round bottom flask of an N, N'-JI [2-(2, 3-dihydroxy propyloxy) ethyl]-N, and N'-JIHEKISA desyl chestnut amide (7-a). this -- dimethyl malonate 0.18g (1.4mmol) -- in addition, it was made to react for 8 to 12 hours, the silica gel column chromatography refined the obtained rough product (expansion solvent chloroform: methanol = 20:1-5:1), and 0.25g was obtained as a viscous paste-like solid-state (7-a) When the obtained compound (7-a) was observed with the polarization microscope, it showed the multiple color type polarization image by the birefringence. Moreover, the physical properties are as follows.

[0048] 1 H-NMR delta(CDCl3) = 0.88 (6H, t, J= 7Hz), 1.1-1.4 (52H, broad s), 1.4-1.7 (4H, broad m), 3.2-4.0(26H) IR(cm-1):3384, 2920, 1630, 1462, 1118, 1044, 910, 724. [0049] The emulsification constituent for makeup was prepared by the combination shown in Table 1 using the glyceryl ether-ized amide compound (7-a) obtained in the example 2 example 1. [0050]

[Table 1]

	調製直後	5℃で2日間保存後
実施例2	クリーム状(粘度5500mPa'S. 30℃)	相分離、結晶析出は認められず、 安定な乳化状態を維持。
実施例3	乳液状(粘度980mPa'S。30℃)	相分離、結晶析出は認められず、 安定な乳化状態を維持。
比較例1	硬いゲル状	多量の不定形結晶が折出

[0058] From Table 3, when the emulsification constituent for makeup of this invention blended the glyceryl ether-ized amide compound of this invention, as compared with what blended the alcoholic conventional amide compound, the bird clapper was checked as an emulsification state is stable. Rather than before, the emulsification constituent for makeup by combination of Table 1 and 2 is the amount of a surfactant and an oily medicine reduced sharply, and forms the stable emulsification system. [0059] Degreasing processing by the acetone ether was performed to 15 example of examination 2 subjects' right-and-left forearm inside section, and it considered as the model of the artificial-drying dry-area skin. Subsequently, the five above-mentioned subjects were divided into each three groups, the emulsification constituent for makeup of an example 2, an example 3, and the example 1 of comparison was applied to the left arm inside section which performed degreasing processing for three days and three days, respectively, and the moisture recovery factor of a skin horny layer was evaluated by measuring skin conductance. Here, a moisture recovery factor shall perform degreasing processing by the acetone ether, shall make the skin conductance of the unsettled section 100% for the skin conductance of the right arm inside section of a request 0%, and shall show it by the average of the rate of increase of the skin conductance of the left arm inside section. A result is shown in Table 4.

[Table 4]

	塗布3日後の皮膚コンダクタンス				堂布3日後の水	
	被験者	右腕	左腕	未処理部	分回復率(%)	平均億(%)
実施例 2	Α	. 8	25	26	94	
	В	6	23	28	77	85
	С	11	. 28	28	100	
	D	. 8	20	26	75	
	E	10	24	28	78	
実施例 3	F	12	21	24	25	
	G	-7	19	28	57	1
	H	9	18	25	44	63
	1	7	. 23	23	100	
	J	7	22	24	88	
比較例 1	K	6	20	28	64	
	L	9	18	26	53	
	М	7	14	27	35	54
	N	6	19	25	68	
	0	8	17	26	50	

[0061] It was checked from Table 4 that the emulsification constituent for makeup of this invention is excellent in skin moisture maintenance capacity.

[0062]

[Effect of the Invention] The glyceryl ether-ized amide compound of this invention is excellent in the skin improvement effects, such as the skin moisture maintenance effect of having excelled extremely, and the rough skin improvement effect, and combination stability. Moreover, the medicine-for-external-application constituent containing this compound has the above-mentioned skin improvement effect, even if it reduces the kind and amount of a surfactant or an oily medicine further, it forms a stable emulsion, and it serves as a feel which felt refreshed by hypoviscosity more.

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#### PRIOR ART

[Description of the Prior Art] In the alcohols which have amide combination, a compound useful as a charge component of makeup exists mostly. For example, the following general formula (2) [0003]

[Formula 2]

[0004] (The inside Ra of a formula is a hydrocarbon group, and Rb -Rh is a hydrogen atom or a methyl independently respectively.) Ri And Rj It is the hydrocarbon group which has a hydrogen atom or a carbon atom to 24 pieces independently respectively. X1 And X2 It is a hydrogen atom or a hydroxyl independently, and is Y1. And Y2 They are a hydrogen atom or a hydroxyl. Y1 And Y2 At least one side is a hydroxyl and Ri. And Rj The united carbon atom Z does not need to exist. Rg And Y2 the united carbon atom W -- it is not necessary to exist (\*\*\*\*\*\* No. 502058 [ eight to ] official report) -- the compound expressed In the water permeability of the skin, play an important role, the intensity of skin structure is made to increase like a ceramide, a ceramide derivative, or a SHUDO ceramide, loss of water is decreased, and it is it. It is considered that it makes the state of the skin improve.

[0005] The compound expressed especially with a general formula (2) has the outstanding emulsification, gelling, and a thickening property, and it is supposed that it is suitable [ for using it in the constituents (for example, cream which has the lotion which has the viscosity of 4000 - 10000 mPa-s, the liquid cream which has the viscosity of 10000 - 20000 mPa-s, 10000 - 20000 mPa-s, or the viscosity beyond it) of the form of a viscous emulsion or gel ] of the compound.

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#### TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] However, the ceramide, the ceramide derivative, the SHUDO ceramide, etc. were not what can not necessarily be satisfied about the skin improvement effect especially the skin moisture maintenance effect, and the rough skin improvement effect, although the thing of the former many had been compounded and used.

[0007] Moreover, a crystal deposits by the cold storage, phase separation arises, and the medicine-for-external-application constituent with which the melting point is comparatively high (the melting point of a compound expressed with a general formula (2) is about 63-85 degrees C), and the ceramide, the ceramide derivative, the SHUDO ceramide, etc. generally contained this may be unable to form a stable emulsification system or stable gel.

[0008] Therefore, this invention aims at offering the compound which was excellent in the skin improvement effect especially the skin moisture maintenance effect, the rough skin improvement effect, etc., and was further excellent in combination stability, and the medicine-for-external-application constituent containing this compound.

octadecyl, a nona desyl, Heneicosyl, DOKOSHIRU, nona KOSHIRU, a thoria contest chill, isostearyl, Iso heptadecyl, 2-ethylhexyl, 1-ethyl heptyl, 8-heptadecyl, 8-heptadecenyl, 8, a 11-heptadeca diethyl, 2-heptyl undecyl, 9-octadecenyl, etc. are mentioned.

[0017] The following compounds can be illustrated as a glyceryl ether-ized amide compound of this invention. [0018]

[Formula 4]

(式中Rは炭素数10.12.14.16または18のアルキル基を示す)

[0019] [Formula 5]

## [0022] [Formula 8]

$$C_{16}H_{33}$$
  $0$   $0H$   $0H$   $0H$ 

[0023] Although especially the manufacture method of the glyceryl ether-ized amide compound (1) of this invention is not limited, it is compoundable with the process expressed with the following reaction formula, for example. The glyceryl ether-ized amide compound of this invention is divided into the manufacture method 1 and the manufacture method 2 by whether it has a hydroxyl at beta grade of the amino group of raw material amines.

[0024] The manufacture method 1, [0025]

[Formula 9]

[0032] It comes out, and like the compound expressed, when a glyceryl ether-ized amide compound (7-1) is unsymmetrical, it is desirable to manufacture a corresponding compound (5-1) separately, and to manufacture a glyceryl ether-ized amide compound (7-1) using these.

[0033] The manufacture method 2, [0034]

[Formula 11]
$$H_2N \longrightarrow 0$$

$$OH \longrightarrow R^7 \longrightarrow 0$$

$$(3-1)$$

$$(4-2)$$

$$R^7$$
OH
 $R^1$ 
OH
 $C = CR^6$ 
 $C = R^8 (6-1)$ 
 $C = CR^6$ 
 $C = CR^6$ 

[0035] (-- R7 shows among a formula the hydrocarbon group of the saturation of the carbon numbers 6-22 which may have branching, or an unsaturation, and R1, R3, R6, and X are the same as the above It is the method expressed with).

[0036] That is, a glyceryl ether-ized amide compound (7-2) is obtained by making an epoxy compound (4-2) react, and making a

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#### **EXAMPLE**

[Example] Although an example is given to below and this invention is further explained to a detail, this invention is not limited to the following examples.

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[0048] 1 H-NMR delta(CDCl3) = 0.88 (6H, t, J= 7Hz), 1.1-1.4 (52H, broad s), 1.4-1.7 (4H, broad m), 3.2-4.0(26H) IR(cm-1):3384, 2920, 1630, 1462, 1118, 1044, 910, 724. [0049] The emulsification constituent for makeup was prepared by the combination shown in Table 1 using the glyceryl ether-ized amide compound (7-a) obtained in the example 2 example 1. [0050]

[Table 1]

成 分 名	%W/W
実施例1のグリセリルエーテル 化アミド化合物(7 - a)	2, 5
スクワラン	2. 0
メチルパラベン	0. 3
ソルピタンモノステアレート	2. 5
ポリオキシエチレン(20)ソルビ タンモノステアレート	1.0
86%グリセリン	4.5
ポリオキシエチレン硬化ヒマシ 油(40B. O. )	1: 0
精製水	100.00まで (バランス)

[0051] The emulsification constituent for makeup was prepared by the combination shown in Table 2 using the glyceryl ether-ized amide compound (7-a) obtained in the example 3 example 1. [0052]

	塗布3日後の皮膚コンダクタンス				空布3日後の水	水分回復率の
	放験者	右腕	左腕	未処理部	分回復率(%)	平均值(%)
実施例 2	A	8	25	26	94	
	В	6	23	28	77	85
	C	11	28	28	100	
	D	8	20	26	75	
Δ	E	10	24	28	78	
実施例3	Ę	12	21	24	25	
·	C	.7	19	28	57	
	Н	9	18	25	44	63
	I	7	. 23	23	100	
	J	7	22	24	88	
比较例1	K	6	20	28	64	
	L	9	18	26	53	
	М	7	14	27	35	54
	N	6	19	25	68	
	0	8	17	26	50	

[0061] It was checked from Table 4 that the emulsification constituent for makeup of this invention is excellent in skin moisture maintenance capacity.

[0062]